Effect of Temperature on Excess Molar Volumes and Viscosities for Propylene Carbonate + *N*,*N*-Dimethylformamide Mixtures

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Densities and viscosities have been determined as a function of mole fraction for propylene carbonate + *N*,*N*-dimethylformamide mixtures at 298.15, 308.15, 318.15, 328.15, and 338.15 K. From these measurements, the excess molar volume (V_m^E) and deviation in viscosity ($\Delta\eta$) were calculated. These results were fitted to the Redlich−Kister polynomials. The values of V_m^E are found to be positive, whereas those of $\Delta\eta$ are negative over the entire composition range at each temperature investigated. The values of V_m^E decrease but those of $\Delta\eta$ increase with increasing temperature. Results have been explained on the basis of molecular interactions in the mixtures.

Introduction

A thorough knowledge of the thermodynamic and transport properties of multicomponent liquid systems is essential in many industrial applications, such as design calculation, heat transfer, mass transfer, fluid flow, and so forth (Venkatesu and Rao, 1997). It is easy to understand the importance of the availability of the solvent thermodynamic parameters, such as density (ρ) and viscosity (η), which are two important physical properties of solvent systems and are often used to explain the medium effects of solvent on transport phenomena, electrolyte behavior, and reaction mechanisms in solutions (Muhuri and Hazra, 1995). As is well-known, these properties are functionally dependent on temperature and, at least for binary mixtures, on the composition of the solvent systems (Marchetti et al., 1991). The changes of these parameters with temperature and mole fraction often provide more and more detailed evidence about the structural character of the systems themselves.

Propylene carbonate (PC), a dipolar aprotic solvent, has been assumed to be an "ideal structureless dielectric" solvent for studies of electrolytes (Friedman, 1967; Cogley et al., 1971; Muhuri et al., 1993) because of its high dielectric constant (64.40 at 298.15 K) and dipole moment (4.94 D). The chemical properties of this solvent have given it an important place in the development of high-energy batteries (Jasinski, 1970; Payne and Theodorou, 1972). Some lithium batteries have adopted mixed organic electrolytes containing PC, because the solvent mixtures present improved electrical properties compared with a pure solvent. Therefore, studies of the thermodynamic and transport properties of mixtures containing PC should be very interesting.

N,*N*-Dimethylformamide (DMF) is a very good aprotic protophilic medium for organic and inorganic substances (Marchetti et al., 1991). It is one of the most important solvents in analytical chemistry and for practical purposes. Research on lithium batteries has found that the conductivity of lithium salts in PC + amide mixed solvents was higher than that in PC alone. So PC + DMF mixtures have potential use in lithium batteries (Tobishima et al., 1988).

There have been some reports on density, viscosity, and excess properties for PC + 2-methoxyethanol (Muhuri and Hazra, 1995), PC + liner and cyclic ketones (Comelli and Francesconi, 1995), PC + dimethyl carbonate, PC + diethyl carbonate (Francesconi and Comelli, 1995), PC + tetrahydrofuran, PC + methanol (Muhuri et al., 1996), PC + acetonitrile (Moumouzias et al., 1991), PC + 1,2-dimethoxyethane (Muhuri and Hazra, 1994), and PC + dimethyl sulfoxide (Ritzoulis, 1989) mixtures. To the best of our knowledge, there have been no density and viscosity data for PC + DMF systems in the literature. In the present work, the densities and viscosities have been measured for PC + DMF mixtures over the entire composition range at 298.15, 308.15, 318.15, 328.15, and 338.15 K. From these data, the excess molar volume V_m^E and the deviation in viscosity $\Delta\eta$ have been calculated and fitted to the Redlich−Kister polynomials. Results have been discussed briefly on the basis of molecular interactions in the mixtures.

Experimental Section

Materials. Propylene carbonate (Luoyang Chemical Factory, A.R.) was dried over molecular sieves (4A) for several days, refluxed for 5 h in the presence of sodium (Na), and then distilled at reduced pressure three times, with the middle fraction of the distillate being collected. *N*,*N*-Dimethylformamide (Tianjin Chemical Factory, A.R.) was mixed with 10% (by volume) benzene (Beijing Chemical Factory, A.R.), and the azeotrope was distilled off under atmospheric pressure at about 353 K. The product was dried over silica gel and distilled at reduced pressure, with the middle fraction being collected. Both the purified solvents were stored over P_2O_5 in a desiccator before use. The solvents were analyzed by Karl−Fischer titration; the water content was <100 ppm.

Density Measurements. Mixtures were prepared by mass on the molality concentration scale. Solution densities were measured with a vibrating-tube digital density meter (model DMA 60/602, Anton Paar, Austria). The accuracy of the density is estimated to be $\pm 1.5 \times 10^{-5}$ g·cm^{-3}. The temperature around the density meter cell was controlled by circulating water from a constant-temperature bath (Schott, Germany). A CT1450 temperature controller and a CK-100 ultracryostat were employed to maintain the bath

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10.1021/je9902746 CCC: $19.00

Table 1. Comparison of the Experimental Density (ρ) and Absolute Viscosity (η) with Literature Values for PC and DMF at Various Temperatures

	ρ/(g·cm^{-3})		η/(mPa·s)		ρ/(g·cm^{-3})		η/(mPa·s)	
T/K	lit.	exptl	lit.	exptl	lit.	exptl	lit.	exptl
	PC				DMF			
298.15	1.199 3[a]	1.199 27	2.513[a]	2.508	0.943 89[e]	0.944 04	0.8012[e]	0.802
	1.198 83[b]		2.4712[b]		0.943 93[f]		0.8136[g]	
	1.198 9[c]		2.508[c]		0.945 60[g]		0.7912[h]	
308.15	1.189 70[b]	1.188 64	2.0476[b]	2.075	0.935 81[g]	0.934 49	0.7166[h]	0.711
	1.189 25[d]						0.7217[g]	
318.15	1.177 96[b]	1.178 14	1.7234[b]	1.746	0.926 28[g]	0.924 86	0.6460[g]	0.635
	1.178 67[d]							
328.15	1.168 17[d]	1.167 57		1.494	0.916 7[g]	0.915 42	0.5843[g]	0.574
338.15	1.157 71[d]	1.157 10		1.301	0.907 03[g]	0.906 06	0.5321[g]	0.525

[a] Jansen and Yeager (1973). [b] Muhuri et al. (1996). [c] Hanna and Sudani (1987). [d] Barthel et al. (1995). [e] Chittleborough et al. (1988). [f] Awwad et al. (1988). [g] Marchetti et al. (1991). [h] Osinska et al. (1983).

temperature to within ±0.005 K. The density meter was calibrated once a day with doubly distilled water and dry air at each temperature concerned. The densities of water at different temperatures were selected from the literature (Kell, 1975). The density of air was calculated according to the formula

$$\rho_a = 0.001\,293\,0 \times p/(1 + 0.003\,67 \times t)/760 \qquad (1)$$

where p is the atmosphere pressure (mmHg) and t is the temperature (°C).

Viscosity Measurements. Solution viscosities were measured with a suspended level Ubbelohde Viscometer, which was placed in a water thermostat (Schott, Germany) and has a flow time of about 200 s for water at 298.15 K. The temperature of the water thermostat was controlled to be as precise as the density measurements. The viscometer was calibrated using the efflux time of water at 298.15 and 308.15 K. The required viscosity data for water were taken from the literature (Weast, 1982−1983). Flow time measurements were performed with a Schott, AVS 310 photoelectric time unit with a resolution of 0.01 s. At least three measurements were made for each solution sample. The absolute viscosity (η) of the solution is given by the following equation:

$$\eta/\rho = ct' - k/t' \qquad (2)$$

where ρ is the solution density, t' is the flow time, and c and k are the cell constants. The estimated accuracy of the experimental viscosity is ±0.3%. Comparison of values of experimental densities and viscosities for pure components with literature data are made in Table 1 at various temperatures.

Results and Discussion

Experimental values of density and absolute viscosity at five temperatures were collected in Table 2 for PC, DMF, and 18 of their mixtures in the whole composition range.

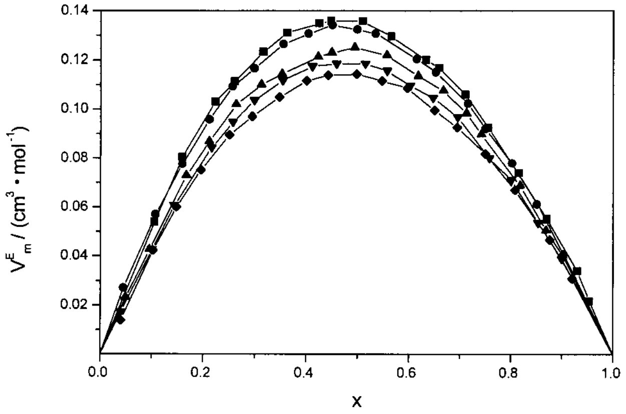

Figure 1. Excess molar volumes V_m^E for xPC + (1 − x)DMF at 298.15 K (■), 308.15 K (●), 318.15 K (▲), 328.15 K (▼), and 338.15 K(◆).

Density and Excess Molar Volumes. The excess molar volumes have been calculated by the following equation:

$$V_m^E = [xM_1 + (1 - x)M_2]/\rho - xM_1/\rho_1 - (1 - x)M_2/\rho_2 \qquad (3)$$

where ρ_1, ρ_2, and ρ are the densities of PC, DMF, and their mixtures at the given temperature, respectively. M_1 and M_2 are the molar masses of the two pure components PC and DMF, and x is the mole fraction of PC in the mixtures. The excess molar volumes V_m^E of the binary mixtures at five temperatures are also listed in Table 2. The composition dependence of V_m^E has been analyzed by least-squares fitting to the Redlich−Kister polynomial:

$$V_m^E/(\text{cm}^3\cdot\text{mol}^{-1}) = x(1 - x)\sum_{k\geq 0} a_k(2x - 1)^k \qquad (4)$$

The coefficients a_k of eq 4 are given in Table 3, together with the standard deviations of the regressions, defined by

$$\delta(V_m^E) = \left[\frac{\sum[(V_m^E)_{\text{exptl}} - (V_m^E)_{\text{calcd}}]^2}{(N - M)}\right]^{0.5} \qquad (5)$$

where N is the total number of experimental points and M is the number of adjustable parameters.

Figure 1 shows the variation of V_m^E as a function of the binary composition at different temperatures. It can be seen that the values of the excess molar volumes are positive in all the ranges of composition for mixtures of PC + DMF, and they decrease with increasing temperature. This is similar to the case for PC + cyclohexanone mixtures (Comelli and Francesconi, 1995). The curves in Figure 1 clearly show the presence of a sharp maximum at each temperature. The maximum becomes less positive and always centered at $x \approx 0.5$ as the temperature increases, confirming the maximum specific interaction at equimolar composition. In interpreting V_m^E in terms of molecular phenomena, positive values are explained by the breaking of chemical or nonchemical interactions among molecules in the pure components during the mixing process (Amigo et al., 1993; Holgado et al., 1993), whereas a more efficient packing in the mixture than in the pure liquids is considered to be the major contribution to the negative V_m^E

Table 2. Experimental Density (ρ), Absolute Viscosity (η), Excess Molar Volume (V_m^E) and Viscosity Deviation ($\Delta\eta$) for xPC + (1 − x)DMF Mixtures as a Function of Composition and Temperature

x	ρ / g·cm^{-3}	η / mPa·s	V_m^E / cm^3·mol^{-1}	$\Delta\eta$ / mPa·s	x	ρ / g·cm^{-3}	η / mPa·s	V_m^E / cm^3·mol^{-1}	$\Delta\eta$ / mPa·s
T = 298.15 K									
0.0000	0.944 04	0.801	0.0000	0.000	0.5658	1.092 62	1.462	0.1296	−0.305
0.1053	0.972 61	0.890	0.0540	−0.091	0.6334	1.109 62	1.582	0.1200	−0.300
0.1593	0.987 03	0.942	0.0805	−0.132	0.6590	1.116 00	1.630	0.1167	−0.295
0.2227	1.003 87	1.004	0.1030	−0.178	0.7108	1.128 88	1.742	0.1059	−0.272
0.2607	1.013 92	1.047	0.1113	−0.200	0.7559	1.140 05	1.835	0.0924	−0.257
0.3155	1.028 29	1.107	0.1233	−0.233	0.8147	1.154 50	1.983	0.0740	−0.209
0.3624	1.040 49	1.164	0.1310	−0.256	0.8705	1.168 08	2.117	0.0553	−0.171
0.4248	1.056 64	1.246	0.1350	−0.280	0.9299	1.182 425	2.284	0.0341	−0.104
0.4476	1.062 50	1.278	0.1360	−0.287	0.9540	1.188 24	2.357	0.0217	−0.0729
0.5094	1.078 28	1.370	0.1359	−0.300	1.0000	1.199 27	2.508	0000	0.000
T = 308.15 K									
0.0000	0.934 49	0.710	0.0000	0.000	0.4988	1.065 48	1.167	0.1323	−0.224
0.0453	0.946 76	0.742	0.0270	−0.0306	0.5351	1.074 67	1.213	0.1308	−0.228
0.1078	0.963 57	0.787	0.0571	−0.0709	0.6158	1.094 97	1.330	0.1204	−0.224
0.1606	0.977 66	0.828	0.0777	−0.102	0.6532	1.104 28	1.380	0.1151	−0.222
0.2114	0.991 10	0.870	0.0958	−0.129	0.7153	1.119 66	1.480	0.1024	−0.207
0.2565	1.002 95	0.910	0.1093	−0.151	0.7499	1.128 19	1.539	0.0921	−0.195
0.2989	1.014 06	0.950	0.1166	−0.169	0.8020	1.140 93	1.638	0.0779	−0.167
0.3551	1.028 64	1.005	0.1266	−0.190	0.8517	1.153 02	1.731	0.0612	−0.142
0.4045	1.041 39	1.058	0.1308	−0.205	0.9033	1.165 51	1.841	0.0408	−0.102
0.4505	1.053 16	1.109	0.1342	−0.216	1.0000	1.188 64	2.075	1.0000	0.000
T = 318.15 K									
0.0000	0.924 86	0.635	0.0000	0.000	0.4941	1.054 23	1.015	0.1251	−0.167
0.0490	0.938 11	0.664	0.0231	−0.025	0.5567	1.070 01	1.081	0.1219	−0.172
0.0961	0.950 78	0.694	0.0427	−0.048	0.6184	1.085 45	1.152	0.1135	−0.170
0.1672	0.969 67	0.740	0.0729	−0.080	0.6678	1.097 68	1.216	0.1076	−0.161
0.2118	0.981 44	0.772	0.0869	−0.099	0.7127	1.108 76	1.271	0.0983	−0.155
0.2639	0.995 07	0.811	0.1019	−0.118	0.7415	1.115 85	1.311	0.0898	−0.148
0.3122	1.007 66	0.849	0.1100	−0.132	0.8179	1.134 47	1.424	0.0688	−0.120
0.3528	1.018 18	0.884	0.1144	−0.143	0.8689	1.146 82	1.505	0.0505	−0.0946
0.4228	1.036 14	0.946	0.1213	−0.159	0.9013	1.154 59	1.560	0.0404	−0.0756
0.4487	1.042 73	0.970	0.1230	−0.163	1.0000	1.178 14	1.746	0.0000	0.0000
T = 328.15 K									
0.0000	0.915 42	0.574	0.0000	0.000	0.5152	1.049 54	0.915	0.1185	−0.133
0.0403	0.926 31	0.595	0.0173	−0.0165	0.5571	1.060 05	0.953	0.1156	−0.134
0.1005	0.942 40	0.629	0.0427	−0.0381	0.6023	1.071 32	0.995	0.1096	−0.133
0.1438	0.953 87	0.651	0.0608	−0.0553	0.6476	1.082 52	1.040	0.1046	−0.130
0.2168	0.973 06	0.695	0.0843	−0.0790	0.6956	1.094 33	1.090	0.0969	−0.123
0.2573	0.983 62	0.721	0.0948	−0.0904	0.7577	1.109 54	1.161	0.0800	−0.110
0.2991	0.994 46	0.748	0.1036	−0.101	0.8006	1.119 92	1.212	0.0712	−0.0980
0.3536	1.008 51	0.787	0.1117	−0.113	0.8530	1.132 58	1.280	0.0537	−0.0789
0.4115	1.023 31	0.830	0.1176	−0.123	0.8997	1.143 76	1.343	0.0389	−0.0588
0.4588	1.035 33	0.867	0.1186	−0.129	1.0000	1.167 57	1.494	0.0000	0.000
T = 338.15 K									
0.0000	0.906 06	0.525	0.0000	0.000	0.4982	1.035 34	0.804	0.1144	−0.107
0.0401	0.916 87		0.0138		0.5475	1.047 66	0.840	0.1116	−0.109
0.1025	0.933 44		0.0424		0.5967	1.059 86	0.880	0.1086	−0.108
0.1481	0.945 46		0.0602		0.6504	1.073 12	0.924	0.0995	−0.106
0.1956	0.957 91		0.0753		0.6949	1.084 02	0.963	0.0926	−0.101
0.2507	0.972 25		0.0896		0.7492	1.097 22	1.013	0.0819	−0.0927
0.2946	0.983 61		0.0972		0.8076	1.111 34	1.075	0.0670	−0.0768
0.3480	0.997 32		0.1049		0.8759	1.127 70	1.146	0.0468	−0.0587
0.4003	1.010 64		0.1116		0.9201	1.139 82	1.197	0.0310	−0.0418
0.4424	1.021 30	0.765	0.1139	−0.103	1.0000	1.157 10	1.301	0.0000	0.000

Table 3. Coefficients a_k of Eq 4 and the Standard Deviations of the Regression at Different Temperatures

T/K	A_0	A_1	A_2	$\delta(V_m^E)$/cm^3·mol^{-1}
298.15	0.5416	−0.0757	0.0079	0.0013
308.15	0.5301	−0.0742	0.0063	0.0007
318.15	0.4969	−0.0433	−0.0209	0.0013
328.15	0.4757	−0.0469	−0.0287	0.0011
338.15	0.4565	−0.0350	−0.0167	0.0014

values. Studies on the dielectric and spectroscopic properties of PC indicate that PC behaves as a normal polar molecule with strong dipole−dipole interaction. Unlike water, PC does not exhibit any strong self-association or well-defined intermolecular structure (Cogley et al., 1971; Muhuri and Hazra, 1994). Because of its large tendency to provide hydrogen bonds via acceptor sites of the amido group −C(O)N=, DMF has weak self-association (Marchetti et al., 1991). Then, in the mixing process, the positive contribution to V_m^E would be due to the breaking of two kinds of interaction: (i) the intermolecular dipolar forces in PC and (ii) the hydrogen bonds presented in self-associated DMF molecules. The positive deviation can also be ascribed to the inefficient packing in the mixture of these components as a result of their incompatible structures (PC, flat circle structure; DMF, chain structure) and the approximate molar volumes (the molar volumes of PC and DMF at 298.15 K are 85.2 cm^3·mol^{-1} and 77.39 cm^3·mol^{-1},

Table 4. Coefficients b_k of Eq 7 and the Standard Deviations of the Regression at Different Temperatures

T/K	B_0	B_1	B_2	$\delta(\Delta\eta)$/mPa·s
298.15	−1.1928	−0.3488	−0.0849	0.0023
308.15	−0.8948	−0.2506	−0.0696	0.0013
318.15	−0.6752	−0.1728	−0.0406	0.0009
328.15	−0.5289	−0.1291	−0.0202	0.0005

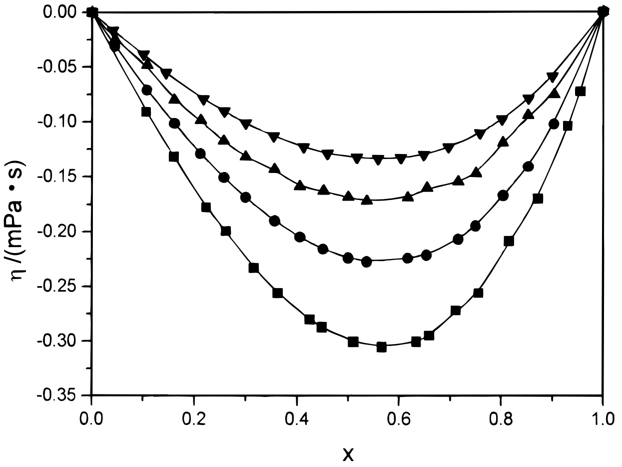

Figure 2. Deviation in viscosity $\Delta\eta$ for xPC + (1 − x)DMF at 298.15 K (■), 308.15 K (●), 318.15K (▲), and 328.15 K (▼).

respectively). These might not allow the components to fit into each others' structures. On the other hand, the existence of hydrogen bonds and dipole−dipole interaction between unlike molecules should be the main contribution to the negative V_m^E. The actual value of V_m^E would depend on the balance of the two opposite contributions. The experimental values indicate that the positive contribution predominates in this mixture.

However, in some systems of PC with cosolvents, the excess molar volumes have exhibited negative deviation from ideality (Francesconi and Comelli, 1995; Muhuri et al., 1996). The system of PC + tetrahydrofuran (THF) is a typical representative (Muhuri et al., 1996). In these systems, dipolar interactions between unlike molecules predominate. This explanation can also be derived from the structure properties and interaction of molecules.

Deviation in Viscosity. The viscosity deviations are obtained by

$$\Delta\eta = \eta_m - x\eta_1 - (1 - x)\eta_2 \qquad (6)$$

where η_m is the viscosity of the mixture and η_1 and η_2 are the viscosities of the pure components PC and DMF, respectively. The composition dependence of $\Delta\eta$ was analyzed by least squares fitting to the equation:

$$\Delta\eta/(\text{mPa}\cdot\text{s}) = x(1 - x)\sum_{k\geq 0} b_k(2x - 1)^k \qquad (7)$$

The values of the coefficient b_k of eq 7 are listed in Table 4 along with the standard deviations of the regressions. The plots of $\Delta\eta$ against the mole fraction of PC are given in Figure 2.

It can be seen from Figure 2 that the deviation in viscosity for the mixtures shows a clear trend: all values of excess viscosity at different temperatures are negative and become less and less negative as the temperature increases, and all minima centered at $x \approx 0.5-0.6$. This result is similar to that for PC + 2-methylmethane mixtures (Muhuri and Hazra, 1995). The deviation in viscosity ($\Delta\eta$) for PC + DMF mixtures is essentially due to two factors: (i) the depolymerization of the associated entities such as PC and DMF to monomeric moieties on mixing, which leads to a negative contribution to $\Delta\eta$, and (ii) the replacement of like contact in pure PC and DMF by unlike contacts, which is responsible for positive values. Therefore, it seems that the former is the main cause for the negative values of $\Delta\eta$ observed in the present work, and no obvious association between PC and DMF would be present in the mixtures. This is in accordance with the V_m^E results.

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Received for review October 8, 1999. Accepted January 17, 2000. We wish to acknowledge the financial support from the National Natural Science Foundation of China (29973009).

JE9902746